EFFECT OF NANO SILICA ON THE COMPRESSIVE STRENGTH OF CONCRETE

SATYAJIT PARIDA
111CE0045
NIT Rourkela

Effect of Nano Silica on the Compressive Strength of Concrete

A Thesis Presented

by

Satyajit Parida

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Prof. Robin Davis P.

Department of Civil Engineering

National Institute of Technology, Rourkela

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ABSTRACT

The application of nanotechnology in concrete has added a new dimension to the efforts to improve its properties. Nanomaterials, by virtue of their very small particle size can affect the concrete properties by altering the microstructure. This study concerns with the use of nano silica of size 236 nm to improve the compressive strength of concrete. An experimental investigation has been carried out by replacing the cement with nano silica of 0.3%, 0.6% and 1% b.w.c. The tests conducted on it shows a considerable increase in early-age compressive strength and a small increase in the overall compressive strength of concrete. The strength increase was observed with the increase in the percentage of nano silica. The FESEM micrographs support the results and show that the microstructure of the hardened concrete is improved on addition of nano silica.

Keywords*:* *concrete, nano silica, compressive strength, microstructure*

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Satyajit Parida

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To friends and family…

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Concrete is the material of present as well as future. The wide use of it in structures, from buildings to factories, from bridges to airports, makes it one of the most investigated material of the 21st century. Due to the rapid population explosion and the technology boom to cater to these needs, there is an urgent need to improve the strength and durability of concrete. Out of the various materials used in the production of concrete, cement plays a major role due its size and adhesive property. So, to produce concrete with improved properties, the mechanism of cement hydration has to be studied properly and better substitutes to it have to be suggested. Different materials known as supplementary cementitious materials or SCMs are added to concrete improve its properties. Some of these are fly ash, blast furnace slag, rice husk, silica fumes and even bacteria. Of the various technologies in use, nano-technology looks to be a promising approach in improving the properties of concrete.

1.1.1 CEMENT- Composition and Hydration

Cement can be described as a crystalline compound of calcium silicates and other calcium compounds having hydraulic properties (Intht). The four major compounds that constitute cement (Bogue's Compounds) are Tricalcium silicate, abbreviated as C_3S, Dicalcium silicate (C_2S), Tricalcium aluminate (C_3A), Tetracalcium aluminoferrite (C_4AF) where C stands for CaO, S stands for SiO_2, A stands for Al_2O_3 and F for Fe_2O_3. Tricalcium silicate and dicalcium silicate are the major contributers to the strength of cement, together constituting about 70 % of cement. Dry or anhydrous cement does not have adhesive property and hence cannot bind the raw materials together to form concrete. When mixed with water chemical reaction takes place and is referred to as 'hydration of cement'. The products of this exothermic reaction are C-S-H gel and $Ca(OH)_2$. Calcium hydroxide has lower surface area and hence does not contribute much to the strength of concrete. On hydration of cement aluminates a product is formed known as ettringite, which has needle like morphology and contributes to some early strength of concrete.

C-S-H gel refers to calcium silicate hydrates, making up about 60 % of the volume of solids in a completely hydrated cement paste. It has a structure of short fibres which vary from crystalline to amorphous form. Owing to its gelatinous structure it can bound various inert materials by virtue of Van der Waal forces. It is the primary strength giving phase in cement concrete.

1.1.2 NANOMATERIALS- Use in Concrete

Nanomaterials are very small sized materials with particle size in nanometres. These materials are very effective in changing the properties of concrete at the ultrafine level by the virtue of their very small size. The small size of the particles also means a greater surface area (Alireza Naji Givi, 2010). Since the rate of a pozzolanic reaction is proportional to the surface area available, a faster reaction can be achieved . Only a small percentage of cement can be replaced to achieve the desired results. These nanomaterials improve the strength and permeability of concrete by filling up the minute voids and pores in the microstructure.

The use of nanosilica in concrete mix has shown results of increase in the compressive, tensile and flexural strength of concrete. It sets early and hence generally requires admixtures during mix design. Nano-silica mixed cement can generate nano-crystals of C-S-H gel after hydration. These nano-crystals accommodate in the micro pores of the cement concrete, hence improving the permeability and strength of concrete.

1.2 MOTIVATION OF THE STUDY

The increased use of cement is essential in attaining a higher compressive strength. But, cement is a major source of pollution. The use of nanomaterials by replacement of a proportion of cement can lead to a rise in the compressive strength of the concrete as well as a check to pollution. Since the use of a very small proportion of Nano SiO_2 can affect the properties of concrete largely, a proper study of its microstructure is essential in understanding the reactions and the effect of the nanoparticles. The existing papers show the use of admixtures in concrete mix. In the present study, no admixture has been used in order to prevent the effect of any foreign material on the strength of the concrete. This study is an attempt to explain the impact of a nano-silica on the compressive strength of concrete by explaining its microstructure.

1.3 OBJECTIVE OF THE STUDY

The main objectives of the present study are as mentioned below:

- To study the effect of nano-silica on the compressive strength of concrete.
- To study the microstructure of the hardened cement concrete.
- To explain the change in properties of concrete, if any, by explaining the microstructure.

1.4 SCOPE OF WORK

The present study incorporates mix design based on the guidelines as per Indian Standard code IS 10262-2009. The nano-silica used is imported from a supplier. The use of any kind of admixture is strictly prohibited in the mix design. The water content has been kept constant to facilitate a better comparison for different samples. The compressive strength measurements are carried out for 7-day and 28-day and the FESEM analysis has been done for 28-day only. The size of the nano-silica was identified using Particle Size Analyser.

1.5 ORGANIZATION OF THE THESIS

This thesis has been organised into five chapters as shown below:

i. The first chapter is the 'Introduction' which gives an idea of the theory involved and the importance of the present work. This is the ongoing chapter.

ii. A 'Review of Literature' follows this chapter which gives an understanding of the various work carried on this field by different authors.

iii. The third chapter, 'Materials and Methods' explains all the material properties and methods used in the experiment.

iv. The fourth chapter, 'Experimental Evidence and Microstructure Analysis' deals with the experimental results of various tests carried on concrete, the FESEM analysis, the PSA analysis and a comparative analysis of the results with the help of tables and graphs.

v. The last chapter, 'Conclusion and Discussion' summarizes the results and interpretations of the study and also states the limitations of the work and the scope for future work.

CHAPTER 2: REVIEW OF LITERATURE

2.1 INTRODUCTION:

In this chapter the works of various authors on the use of nanomaterials in concrete has been discussed in brief. A great number of researches have been performed to understand the nature of nanomaterials and their effect on the properties of concrete. A number of Research & Development work dealing with the use of nanomaterials like Nano silica, colloidal Nano Silica (CNS), Al_2O_3, TiO_2, ZrO_2,Fe_2O_3, carbon nanotubes (CNT) in cement based materials are discussed in the literature. The pozzolanic activity of the material is essential in forming the C-S-H gel and hence the CH crystals are prevented from growing and their number reduces. Thus the early age strength of hardened cement paste is increased. A comparative analysis of this work has been presented in the summary of this chapter which will highlight the significance of each work. Out of the numerous work done in the field only a few relevant works have been highlighted in the next section.

2.2 LITERATURE REVIEW:

H. Li et. al. (2004) experimentally investigated the mechanical properties of nano-Fe_2O_3 and nano-SiO_2 cement mortars and found that the 7 and 28 day strength was much higher than for plain concrete. The microstructure analysis shows that the nanoparticles filled up the pores and the reduced amount of $Ca(OH)_2$ due to the pozzolanic reaction.

Tao Ji (2005) experimentally studied the effect of Nano SiO_2 on the water permeability and microstructure of concrete. The findings show that incorporation of Nano SiO_2 can improve the resistance to water of concrete and the microstructure becomes more uniform and compact compared to normal concrete.

H. Li et.al. (2006) studied the abrasion resistance of concrete blended with nano particles of TiO2 and SiO2 nano particles along with polypropylene (PP) fibers. It was observed that abrasion resistance can be improved considerably by addition of nano particles and PP fibers. Also the combined effect of PP fiber + Nano particles shows much higher abrasion resistance than with nano particles only. It was found that abrasion resistance of nano TiO2 particles is better than nano SiO2 particles. Also relationship between abrasion resistance and compressive strength is found to be linear.

B.-W Jo et. al. (2007) studied the characteristics of cement mortar with Nano SiO_2 particles experimentally and observed higher strength of these blended mortars for 7 and 28 days. The microstructure analysis showed that SiO_2 not only behaves as a filler to improve microstructure, but also as an activator to the pozzolanic reaction.

M.Nill et.al. (2009) studied the combined effect of micro silica and colloidal nano silica on properties of concrete and found that concrete will attain maximum compressive strength when it contains 6% micro silica and 1.5% nano silica. The highest electrical resistivity of concrete was observed at 7.5% micro and nano silica. The capillary absorption rate is lowest for the combination of 3% micro silica and 1.5% nano silica.

Alirza Naji Givi et.al. (2010) studied the size effect of nanosilica particles. They replaced cement with nanosilica of size 15nm and 80nm with 0-5, 1, 1.5 & 2% b.w.c. An increase in the compressive strength was observed with 1.5% b.w.c showing maximum compressive strength. A comparison between particle size showed that for 80nm particles the maximum strength was more than for 15nm particles, also a considerable improvement in flexural and split tensile strength of Nano SiO2 blended concrete was observed.

A. Sadrmotazi et.al. (2010), in another paper, have studied the effect of PP fiber along with nano SiO_2 particles. The nanosilica was replaced up to 7% which improved the compressive strength of cement mortar by 6.49%. PP fiber amounts beyond 0.3% reduces the compressive strength but beyond 0.3% dose of PP fiber increases the flexural strength, showing the effectiveness of nano SiO_2 particles. Also up to 0.5% PP fibers in mortar water absorption decreases which indicates pore refinement.

Ali Nazari et.al. (2010) studied the combined effect of Nano SiO_2 particles and GGBFS on properties of concrete. They used nanosilica with 3% b.w.c. replacement and 45% b.w.c. GGBFS, which shows improved split tensile strength. An improvement in the pore structure of SCC with silica particles was observed. Apart from this hey have studied the effect of ZnO_2 nano particles on SCC concrete with constant w/c ratio of 0.4. The results showed that by increasing the content of super plasticizer flexural strength decreases. Upto 4% b.w.c. of ZnO_2 content an increase in the flexural strength of SCC was recorded. In another experiment the same author studied effect of Al_2O_3 nano particles on the properties of concrete. The results showed that cement could be replaced up to 2% for improving mechanical properties of concrete, but Al_2O_3 nano particles decreased percentage water absorption of concrete. XRD analysis of the sample showed that there is more rapid formation of hydrated product.

M. Collepardi et.al. **(2010)** studied the effect of combination of silica fume, fly ash and ultrafine amorphous colloidal silica (UFACS) on concrete. The result shows that steam cured concrete containing SF and FA alone are much stronger than NC cured at room temperature at early age where as compressive strength at 28-90 days of steam cured concrete is less than NC cured at room temperature. So author advised to use SF,FA&UFACS for the manufacturing of precast unit.

M.S. Morsy et. al. (2010) studied the effect of nano-clay on the mechanical properties and microstructure of Portland cement mortar and observed that the tensile and compressive strength increased by 49% and 7% respectively at 8% nano-metakaolin (NMK).

Surya Abdul Rashid et.al. (2011) worked on the effect of Nano SiO2 particle on both mechanical properties (compressive, split tensile and flexural strength) and physical properties (water permeability, workability and setting time) of concrete which shows that binary blended concrete with nano SiO2 particles up to 2% has significantly higher compressive, split tensile and flexural strength compared to normal concrete. Another inference drawn was that partial replacement of nano SiO2 particles decreases the workability and setting time of fresh concrete for samples cured in lime solution.

Ali Nazari et.al. (2011) studied strength and percentage water absorption of SCC containing different amount of GGBFS and TiO2 nano particles. The findings of the experimentation are that replacement of Portland cement with up to 45% weight of GGBSF and up to 4% weight of TiO2 nano particles gives a considerable increase to the compressive, split tensile and flexural strength of the blended concrete. This increase is due to more the formation of hydrated products in presence of TiO2; also the water permeability resistance of hardened concrete was improved. The author also studied effect of CuO nano particles on SCC and observed that increased percentage of polycarboxylate admixture content results in decreased compression strength. The CuO nano particles of average particle size 15nm content with up to 4% weight increased the compressive strength of SCC. CuO nano particles up to 4% could accelerate the first peak in conduction calorimetric testing which is related to the acceleration of formation of hydrated cement products.

Sekari and Razzaghi (2011) studies the effect of constant content of Nano ZrO2, Fe2O3, TiO2, and Al2O3 on the properties of concrete. The reults showed that all the nano particles have noticeable influence on improvement on durability properties of concrete but the contribution of nano Al2O3 on improvement of mechanical properties of HPC is more than the other nano particles.

A.M. Said et.al. **(2012)** studied the effect of colloidal Nano silica on concrete by blending it with class F fly ash and observed that performance of concrete with or without fly ash was significantly improved with addition of variable amounts of nano silica. The mixture containing 30% FA and 6% CNS provides considerable increase in strength. Porosity and threshold pore diameter was significantly lower for mixture containing Nano silica. The RCPT test shows that passing charges and physical penetration depth significantly improved.

Alireza Naji Givi et.al. (2012) studied the effect of Nano SiO2 particles on water absorption of RHA blended concrete. It is concluded that cement could be replaced up to 20% by RHA in presence of Nano SiO2 particle up to 2% which improves physical and mechanical properties of concrete.

Heidari and Tavakoli (2012) investigated the combined effect of replacement of cement by ground ceramic powder from 10% to 40% b.w.c. and nano SiO2 from 0.5 to 1%. A substantial decrease in water absorption capacity and increase in compressive strength was observed when 20% replacement is done with ground ceramic powder with 0.5 to 1% as the optimum dose of Nano SiO2 particles.

J.Comiletti et.al. (2012) investigated the effect of micro and nano CaCO3 on the early age properties of ultra-high performance concrete (UHPC) cured in cold and normal field conditions. The micro CaCO3 was added from 0 to 15% b.w.c. and nano CaCO3 was added at the rate of 0, 2.5 and 5% b.w.c. Results show that by incorporating nano and micro CaCO3 the flow ability of

UHPC is higher than the control mix which increases the cement replacement level. The mixture containing 5% nano CaCO3 and 15% micro CaCO3 gives shortest setting time at 10 °C and at 20°C the highest 24 hrs compressive strength is achieved by replacing cement with 2.5% nano and 5% micro CaCO3 and highest compressive strength at 26 days was achieved at 0% nano and 2.5% micro CaCO3.

Min. Hong Zhang et.al. (2012) studied the effect of NS & high volume slag mortar on setting time and early strength and observed that rate of hydration increases with addition of NS, compressive strength of slag mortar increases with increase in NS dosages from 0.5 to 2% by weight of cement. 2% NS reduces initial and final setting time and compressive strength increases by 22% and 18% at 3 days and 7 days with addition of 50% slag. NS with particle size 7 & 12 nm are more effective in increasing cement hydration and reaction compared with silica fume.

G. Dhinakaran et. al. (2014) analysed the microstructure and strength properties of concrete with Nano SiO_2. The silica was ground in the planetary ball mill till nano size reached and it was blended in concrete with 5%, 10% and 15% b.w.c.. The experimental results showed gain in compressive strength with maximum strength for 10% replacement.

Mukharjee and Barai (2014) the compressive strength and charcteristics of Interfacial Transition Zone (ITZ) of concrete containing recycled aggregates and nano-silica. An improvement in the compressive strength and microstructure of concrete was observed with the incorporation of nano-silica.

2.3 SUMMARY

The review of a number of literatures shows the importance of this field of research. The findings shows that a number of nanomaterials like SiO_2, TiO_2, Al_2O_3, colloidal nanosilica, metakaolin and others can be incorporated to improve the properties of concrete. The results show the improved characteristics of the blended concrete in terms of compressive, tensile and flexural strength. Apart from that the permeability of the specimen can also be increased by adding a small percentage of the nanomaterial. The SEM, XRD and other analysis shows an improved microstructure with reduced number of pores.

The current study is concerned with the incorporation of Nano SiO_2 only.

CHAPTER 3: MATERIALS AND METHODS

3.1 GENERAL

This chapter is concerned with the details of the properties of the materials used, the method followed to design the experiment and the test procedures followed. The theory is supplemented with a number of pictures to have a clear idea on the methods.

3.2 MATERIAL PROPERTIES

The materials used to design the mix for M25 grade of concrete are cement, sand, coarse aggregate, water and Nano SiO_2. The properties of these materials are presented below.

3.2.1 Properties of Cement

Portland slag cement of 43 grade conforming to IS: 455-1989 is used for preparing concrete specimens. The properties of cement used are given in the Table 2.

Table 3.1: Properties of Portland slag cement

Specific Gravity	Fineness by sieve analysis	Normal consistency
3.014	2.01%	33%

3.2.2 Properties of fine and coarse aggregate

Sand as fine aggregates are collected from locally available river and the sieve analysis of the samples are done. It is found that the sand collected is conforming to IS: 383-1970. For coarse aggregate, the parent concrete is crushed through mini jaw crusher. During crushing it is tried to maintain to produce the maximum size of aggregate in between 20mm to 4.75mm. The coarse aggregate particle size distribution curve is presented in Fig. 3.1. The physical properties of both fine aggregate and recycled coarse aggregate are evaluated as per IS: 2386 (Part III)-1963 and given in Table 3.2.

Table 3.2: Properties of coarse aggregate and fine aggregate

Property	Coarse Aggregate	Fine Aggregate
Specific Gravity	2.72	2.65
Bulk Density (kg/L)	1.408	-
Loose Bulk Density (kg/L)	1.25	-
Water Absorption (%)	4.469	0.0651
Impact Value	26.910	-
Crushing Value	26.514	-
Fineness Modulus	3.38	2.84

3.2.3 Properties of Water

Tap water was used in this experiment. The properties are assumed to be same as that of normal water. Specific gravity is taken as 1.00.

3.2.4 Properties of Nano SiO_2

The average size of nano silica was found to be 236 nm from Particle Size Analyzer, the report of which has been presented in the Appendix. The properties of the material are shown in Table 3.3. Fig. 3. Shows the nano silica used in the experiment.

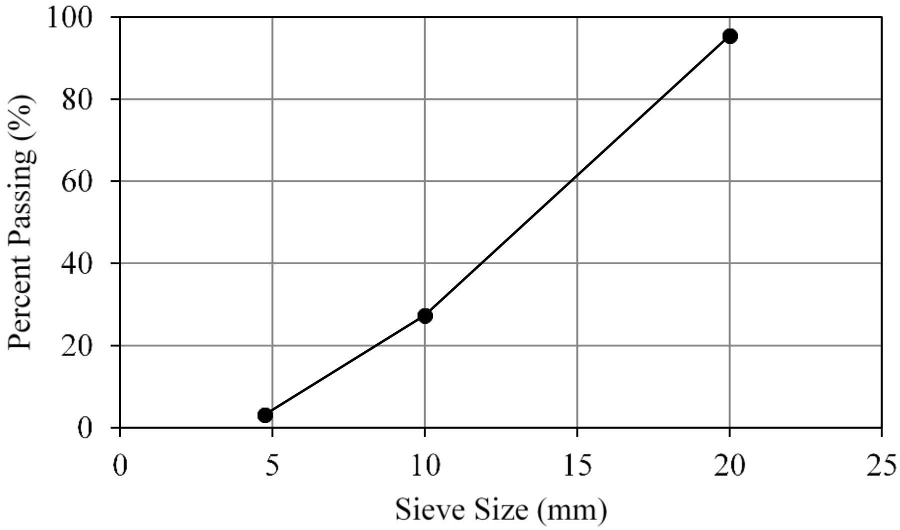


Fig. 3.1: Size distribution curve for coarse aggregate

Fig. 3.2: Image of the Nano SiO_2 used

Table 3.3: Properties of Nano SiO_2

TEST ITEM	STANDARD REQUIREMENTS	TEST RESULTS
SPECIFIC SURFACE AREA (m^2/g)	200 $\pm$ 20	202
PH VALUE	3.7 – 4.5	4. 12
LOSS ON DRYING @ 105 DEG.C (5)	$\leq$ 1. 5	0. 47
LOSS ON IGNITION @ 1000 DEG.C (%)	$\leq$ 2.0	0.66
SIEVE RESIDUE (5)	$\leq$ 0. 04	0. 02
TAMPED DENSITY (g/L)	40 – 60	44
SiO2 CONTENT (%)	$\geq$ 99. 8	99. 88
CARBON CONTENT (%)	$\leq$ 0. 15	0. 06
CHLORIDE CONTENT (%)	$\leq$ 0. 0202	0. 009
Al_2O_3	$\leq$ 0. 03	0. 005
TiO_2	$\leq$ 0. 02	0. 004
Fe_2O_3	$\leq$ 0. 003	0. 001

3.3 METHODS

3.3.1 Mix Design

The mix design for M25 grade of concrete is described below in accordance with Indian Standard Code IS: 10262-1982.

TARGET STRENGTH FOR MIX PROPORTIONING:

Characteristic compressive strength at 28 days: f_{ck} = 25 MPa

Assumed standard deviation (Table 1 of IS 10262:1982): sd = 4 MPa

Target average compressive strength at 28 days: $f_{target} = f_{ck} + 1.65sd$ =31.6 MPa

I. SELECTION OF WATER-CEMENT RATIO:

From Table 5 of IS: 456-2000, maximum water-cement ratio = 0.50

To start with let us assume a water-cement ratio of 0.43

II. SELECTION OF WATER CONTENT:

Maximum water content per cubic metre of concrete (refer Table 2 of IS: 10262-1982): W_{max} = 186L (for 50 mm slump).

Since, the slump was less than 50 mm, no adjustment was required.

III. CALCULATION OF CEMENT CONTENT:

Mass of water selected per cubic metre of concrete = 186 kg.

Mass of cement per cubic metre of concrete = 186/0.43 = 433 kg.

Minimum cement content = 300 kg/m^3 (for moderate exposure condition, Table 5 of IS 456:2000)

Maximum cement content = 450 kg/m^3 (Cl. 8.2.4.2 of IS 456:2000)

So, the selected cement content is alright.

IV. PROPORTION OF VOLUME OF COARSE AGGREGATE AND FINE AGGREGATE CONTENT:

Volume of coarse aggregate per unit volume of total aggregate (Table 3 of IS: 10262-1982) = 0.64

(This is corresponding to 20 mm size aggregate and Zone III fine aggregate for water-cement ratio of 0.50)

As the water-cement ratio is lowered by 0.05, the proportion of volume of coarse aggregate is increased by 0.01 (ref. Table 6 of IS: 10262-1982)

Corrected volume of coarse aggregate per unit volume of total aggregate = (0.64+0.014) = 0.654

Volume of fine aggregate per unit volume of total aggregate = 1-0.654 =0.346

V. MIX CALCULATIONS

i. Volume of concrete = 1 m^3

ii. Volume of cement = 433/(3.01×1000) = 0.144 m^3

iii. Volume of water = 186/1000 = 0.186 m^3

iv. Volume of all aggregates = 1-0.144-0.186 = 0.67 m^3

v. Mass of coarse aggregate = 0.654×0.67×2.72×1000 = 1192 kg

vi. Mass of fine aggregate = 0.346×0.67×2.65×1000 = 614 kg

MIX PROPORTION:

For a batch of 6 cubes of 150mm side, the volume of concrete required

= $(0.15)^3 \times 6 \times 1.2 = 0.024$ m^3 (taking into account 20 % extra for losses)

Cement required	= 0.024x433 = 10.4 kg
Fine aggregate required	= 0.024x614 = 14.7 kg
Coarse aggregate required	= 0.024x1192 = 28.6 kg
Water required	= 0.024x186 = 4.5 kg

3.3.2 Preparation of Test Specimen

For conducting compressive strength test on concrete cubes of size $150 \times 150 \times 150$ mm are casted. A rotary mixture is used for thorough mixing and a vibrator is used for good compaction. After successful casting, the concrete specimens are de-moulded after 24 hours and immersed in water for 28 days maintaining 27 ± 1^0 C. Fig. 3.3 shows some concrete specimen casted in laboratory.

Fig. 3.3 (a): concrete cubes casted in the mould

Fig. 3.3 (b): concrete cubes after de-moulding.

Fig. 3.3: (a) and (b) shows some concrete specimen cast in laboratory

3.3.3 Compressive Strength Test

The compressive strength of specimens is determined after 7 and 28 days of curing with surface dried condition as per Indian Standard IS: 516-1959. Three specimens are tested for typical category and the mean compressive strength of three specimens is considered as the compressive strength of the specified category.

3.3.4 Ultrasonic Pulse Velocity (UPV) Test

It is a non-destructive testing technique (NDT). The method consists of measuring the ultrasonic pulse velocity through the concrete with a generator and a receiver. This test can be performed on samples in the laboratory or on-site. The results are affected by a number of factors such as the surface and the maturity of concrete, the travel distance of the wave, the presence of reinforcement, mixture proportion, aggregate type and size, age of concrete, moisture content, etc., furthermore some factors significantly affecting UPV might have little influence on concrete strength. Table 3.4 shows the quality of concrete for different values of pulse velocity. The images of the UPV Testing Machine used in the laboratory is shown in Fig. 3.4.

Table 3.4: Criteria for quality of concrete

PULSE VELOCITY	CONCRETE QUALITY
>4000 m/s	Excellent
3500-4000 m/s	Very Good
3000-3500 m/s	Satisfactory
<3000 m/s	Poor

3.3.5 Other Tests

Some other tests performed were using Field Emission Scanning Electron Microscope (FESEM) and using Particle Size Analyser (PSA). Since these tests were performed by technical experts, these are not explained here and only the results are presented in the next chapter.

Fig 3.4 (a): UPV Test apparatus

Fig. 3.4 (b): UPV Test of concrete specimen

Fig. 3.4: (a) and (b) shows UPV Test performed in laboratory

CHAPTER 4: EXPERIMENTAL EVIDENCE AND MICROSTRUCTURE ANALYSIS

4.1 GENERAL

This chapter is concerned with the presentation of results of the experiments carried out towards the objective of the project. It includes results from compressive strength test, UPV Test and FESEM. The results are supplemented with graphs in order to have a better analysis of the results.

4.2 EXPERIMENTAL RESULTS

4.2.1 UPV Test Results:

Fig 4.1-4.8 show UPV test results for specimen for 7 day and Fig 4.5-4.8 show UPV test results for specimen for 28 day.

Table 4.1: UPV Test for control specimen for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.10	4678	32.2
2	8.34	4702	31.9
3	8.36	4777	31.4

Table 4.2: UPV Test for specimen with nano-silica 0.3% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.18	4491	33.4
2	8.22	4491	33.4
3	8.24	4386	34.2

Table 4.3: UPV Test for specimen with nano-silica 0.6% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.26	4630	32.4
2	8.08	4630	32.4
3	7.98	4702	31.9

Table 4.4: UPV Test for specimen with nano-silica 1% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.24	4491	33.4
2	8.14	4360	34.4
3	8.30	4559	32.9

Table 4.5: UPV Test for control specimen for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.42	4808	31.2
2	8.36	4854	30.9
3	8.14	4777	31.4

Table 4.6: UPV Test for specimen with nano-silica 0.3% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.06	4673	32.1
2	8.32	4732	31.7
3	8.22	4854	30.9

Table 4.7: UPV Test for specimen with nano-silica 0.6% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.18	4702	31.9
2	8.24	4777	31.4
3	8.22	4777	31.4

Table 4.8: UPV Test for specimen with nano-silica 1% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Velocity (m/s)**	**Time (μs)**
1	8.30	4658	32.2
2	8.30	4702	31.9
3	8.28	4808	31.2

4.2.2 Compressive Strength Test Results

*Compressive Strength = $(52 \times 9.81 \times 1000) \div (150 \times 150) = 22.67$ MPa

Table 4.9: Compressive Strength of control specimen for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.10	52	22.67 *
2	8.34	68	29.65
3	8.36	61	26.59
Mean			26.30

Table 4.10: Compressive Strength of specimen with nano-silica 0.3% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.18	67	29.21
2	8.22	71	30.95
3	8.24	52	22.67
Mean			27.61

Table 4.11: Compressive Strength of specimen with nano-silica 0.6% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.26	66	28.77
2	8.08	72	31.39
3	7.98	76	33.14
Mean			31.1

Table 4.12: Compressive Strength of specimen with nano-silica 1% b.w.c for 7 day

7-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.24	77	33.57
2	8.14	79	34.44
3	8.30	82	35.75
Mean			34.59

Table 4.13: Compressive Strength of control specimen for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.42	84	36.62
2	8.36	84	36.62
3	8.14	75	32.70
Mean			35.31

Table 4.14: Compressive Strength of specimen with nano-silica 0.3% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.06	66	28.78
2	8.32	88	38.37
3	8.22	88	38.37
Mean			35.17

Table 4.15: Compressive Strength of specimen with nano-silica 0.6% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.18	83	36.19
2	8.24	80	34.88
3	8.22	88	38.37
Mean			36.48

Table 4.16: Compressive Strength of specimen with nano-silica 1% b.w.c for 28 day

28-DAY TEST RESULT			
Sample No.	**Weight (kg)**	**Load (tonne)**	**Compressive Strength (MPa)**
1	8.30	88	38.37
2	8.30	93	40.55
3	8.28	93	40.55
Mean			39.82

4.3 COMPARISON OF RESULTS

4.3.1 Comparison of Compressive Strength Results

The change in compressive strength for the blended sample (in %) for 7 and 28 day is shown in Table 4.17 and Table 4.18 respectively. A graphical representation of this result is shown in Fig. 4.1 and Fig. 4.2. The change in compressive strength from 7 day to 28 day is shown in Fig 4.3.

Table 4.17: Comparison of compressive strength for 7 day

7-DAY RESULTS	STRENGTH (MPa)	INCREASE IN STRENGTH (%)
CONTROL	26.30	-
NS 0.3% b.w.c	27.61	4.98
NS 0.6% b.w.c	31.10	18.25
NS 1% b.w.c	34.59	31.52

NS= Nano SiO_2

Table 4.18: Comparison of compressive strength for 28 day

28-DAY RESULTS	STRENGTH (MPa)	INCREASE IN STRENGTH (%)
CONTROL	35.31	-
NS 0.3% b.w.c	35.17	-0.39
NS 0.6% b.w.c	36.48	3.31
NS 1% b.w.c	39.82	12.77

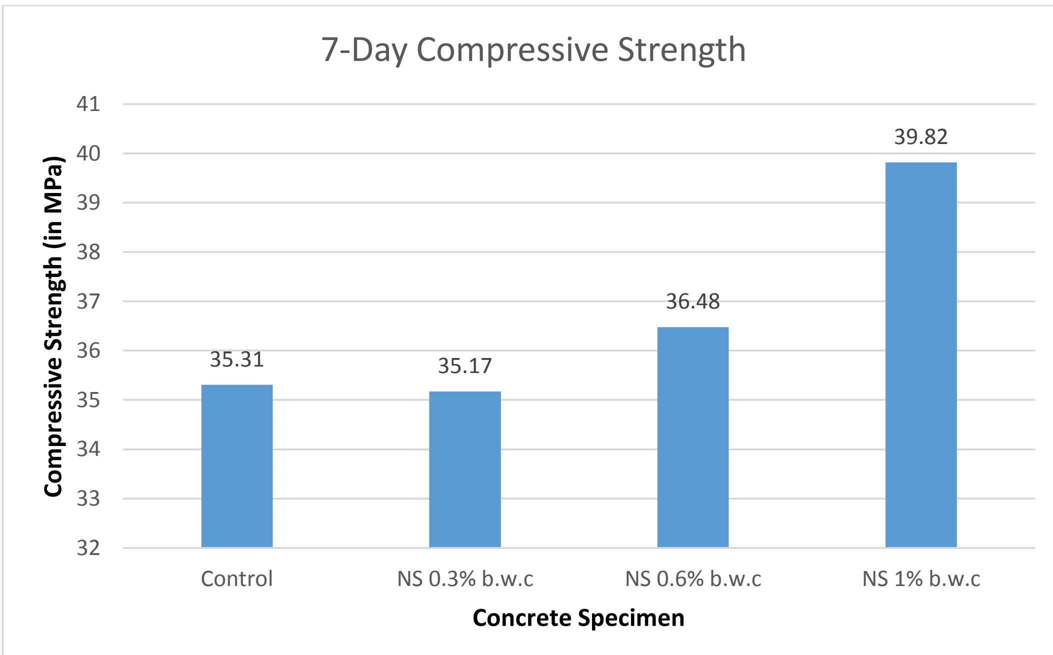


Fig. 4.1: 7-day compressive strength of four specimen

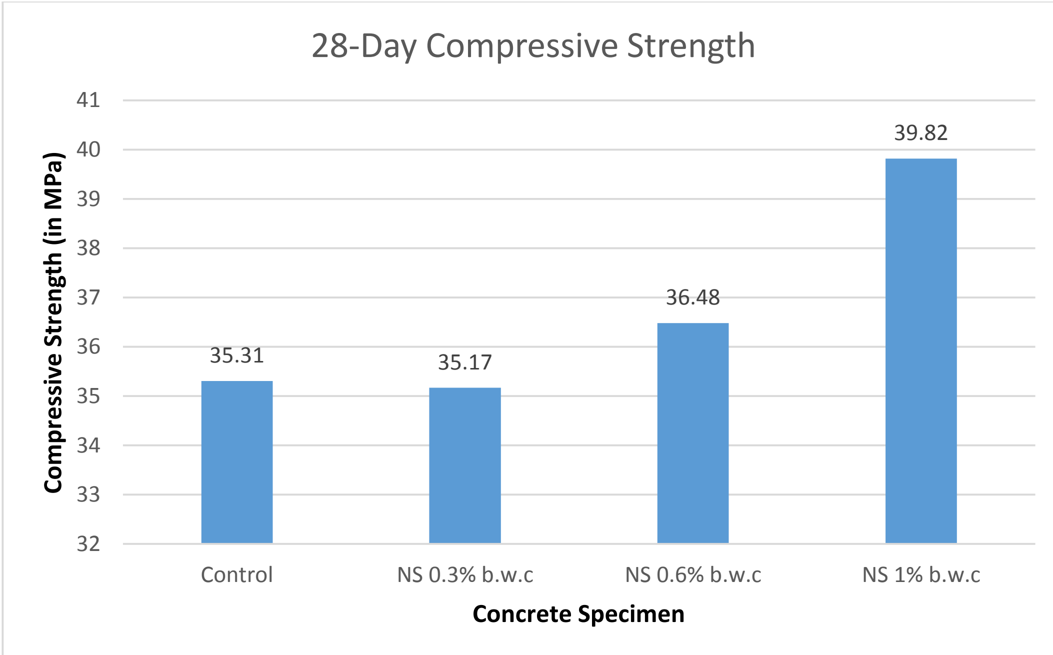


Fig. 4.2: 28-day compressive strength of four specimen

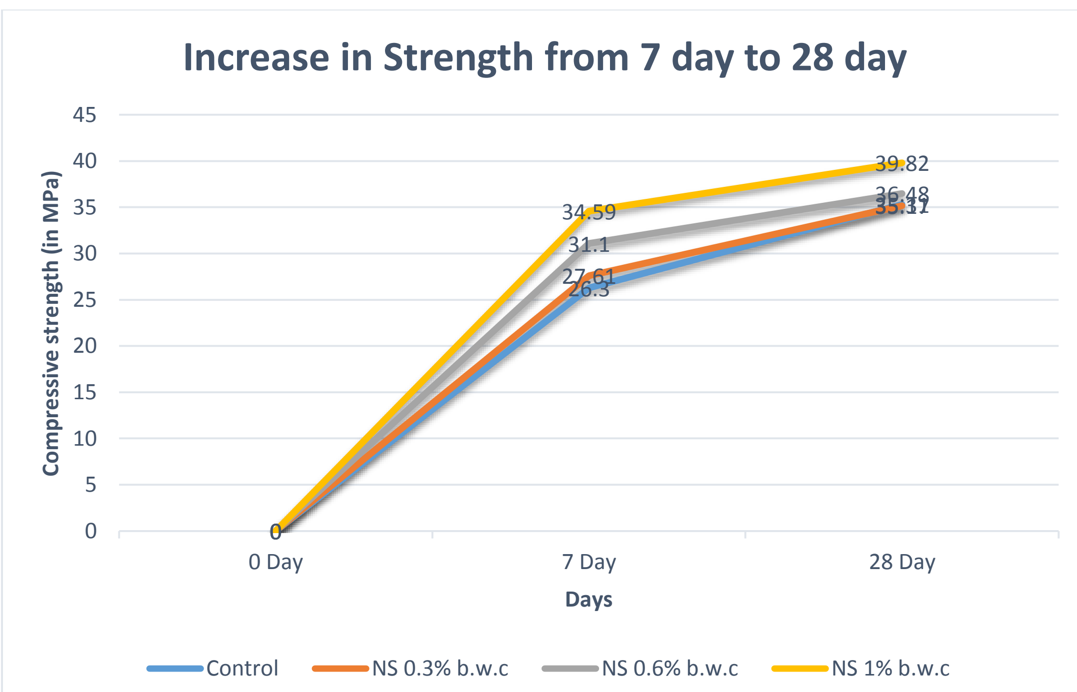


Fig. 4.3: Change in compressive strength of four specimen from 7 day to 28 day

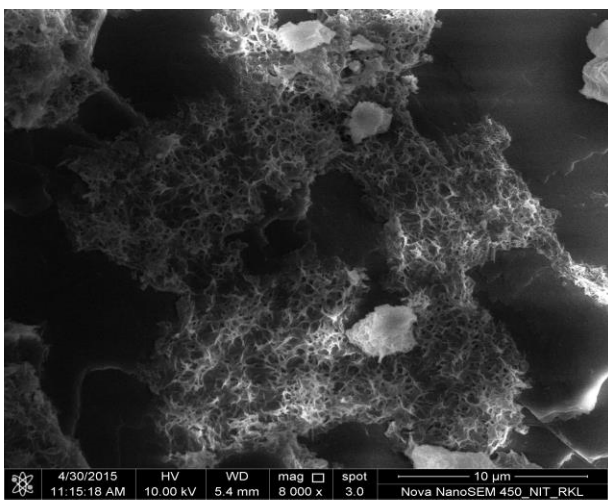
The tables and graphs show that there is an improvement in the early strength of concrete blended with nano silica but later the increase in strength is subdued.

4.3.2 Comparison of UPV Test Results

From the UPV test results, we find that the quality of concrete is very good. The 28-day quality is better than the 7-day quality. The control specimen are found to have better quality compared to the blended concrete specimen.

4.4 Field Emission Scanning Electron Microscope (FESEM) IMAGES

The FESEM micrographs for the four specimens are shown below from Fig 4.4-4.7. Two different magnification has be chosen for the purpose of comparison.



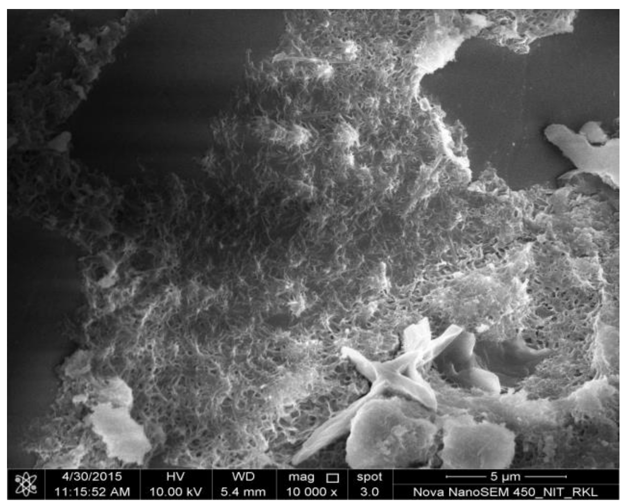


Fig. 4.4: FESEM image of control specimen with different magnification

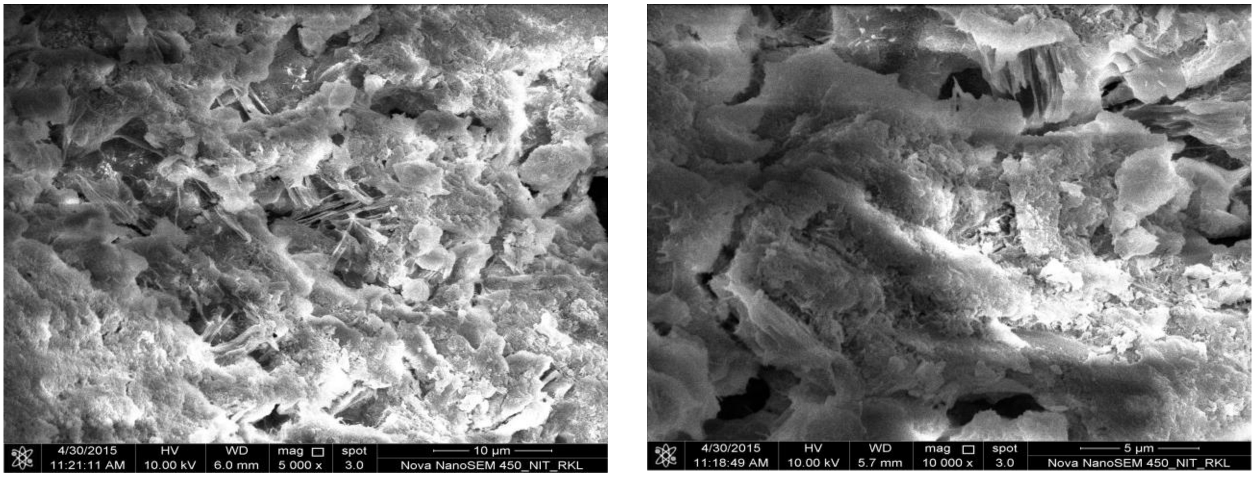


Fig. 4.5: FESEM image of specimen with Nano SiO_2 0.3% b.w.c with different magnificaton

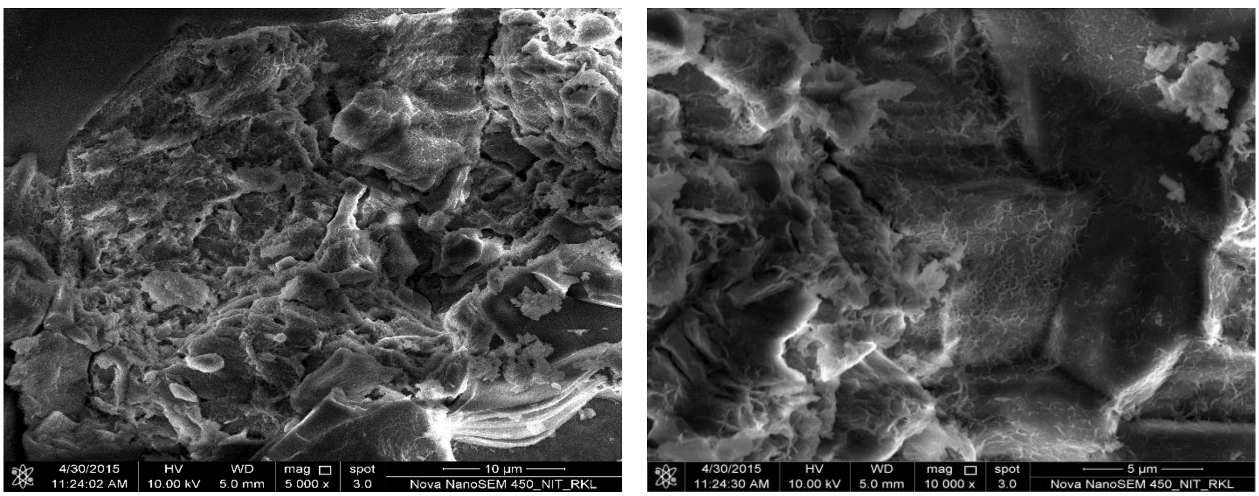


Fig. 4.6: FESEM image of specimen with Nano SiO_2 0.6% b.w.c with different magnificaton

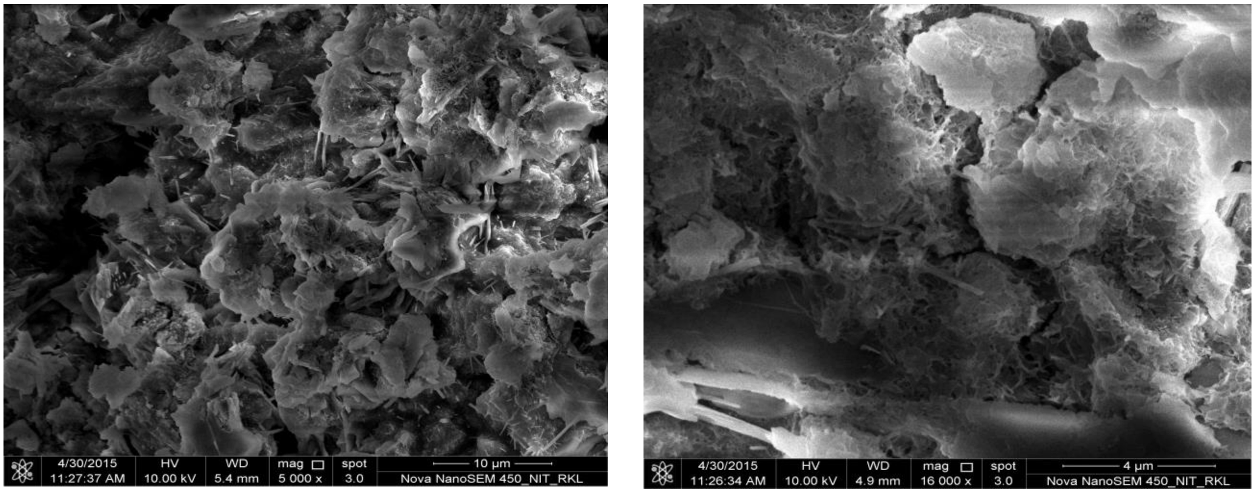


Fig. 4.7: FESEM image of specimen with Nano SiO_2 1% b.w.c with different magnificaton

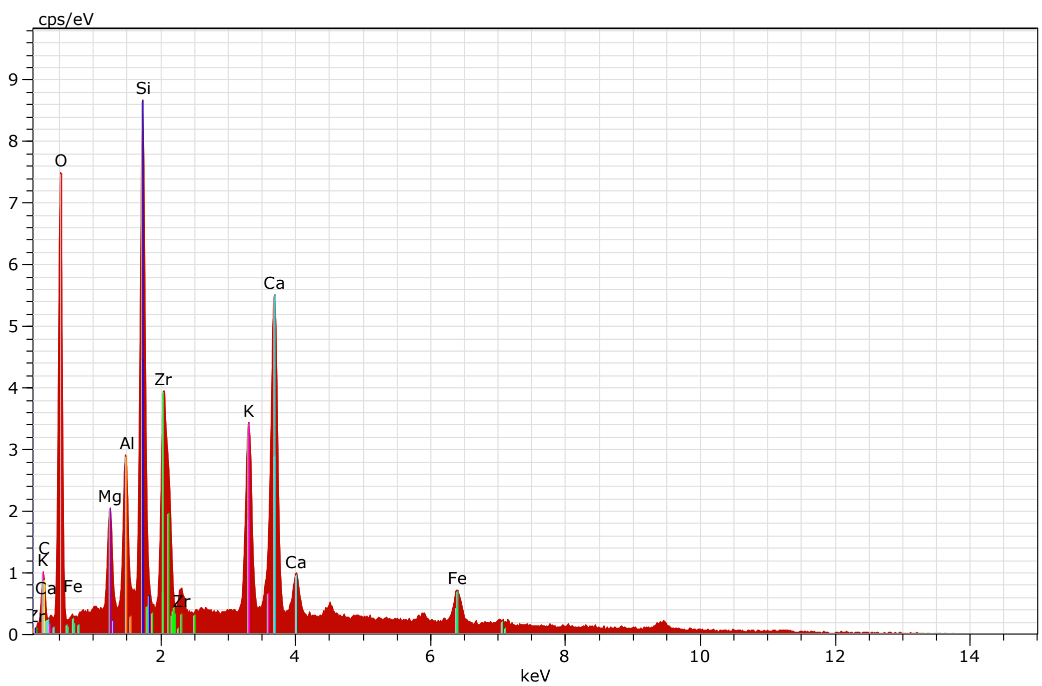


Fig. 4.8: Relative chemical composition for the control specimen

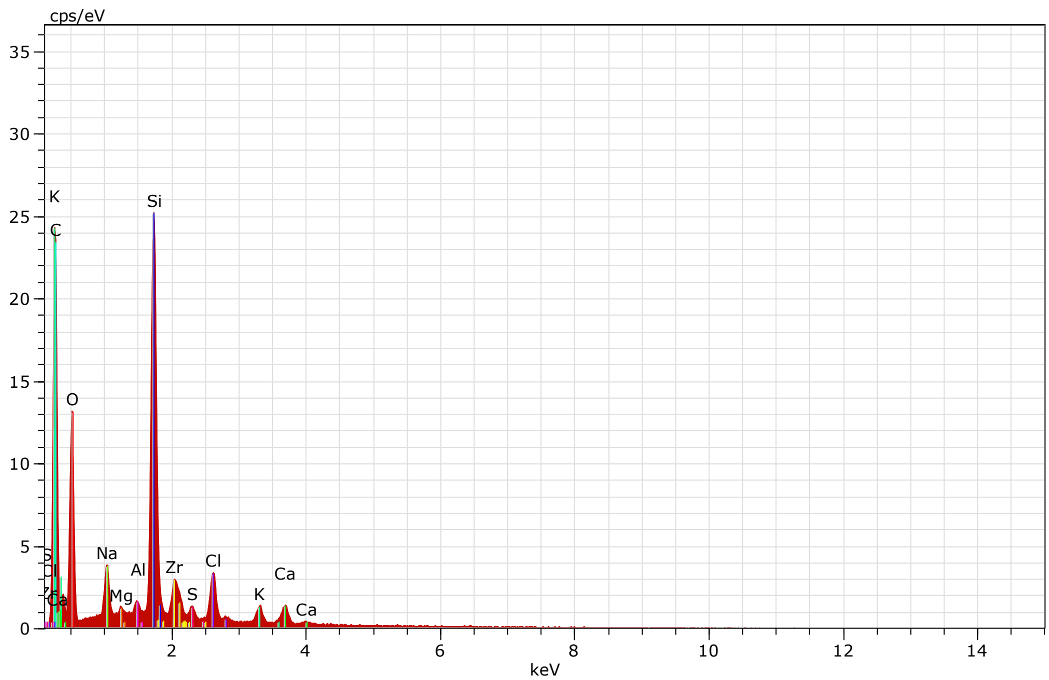


Fig. 4.9: Relative chemical composition for specimen with NS 0.3% b.w.c

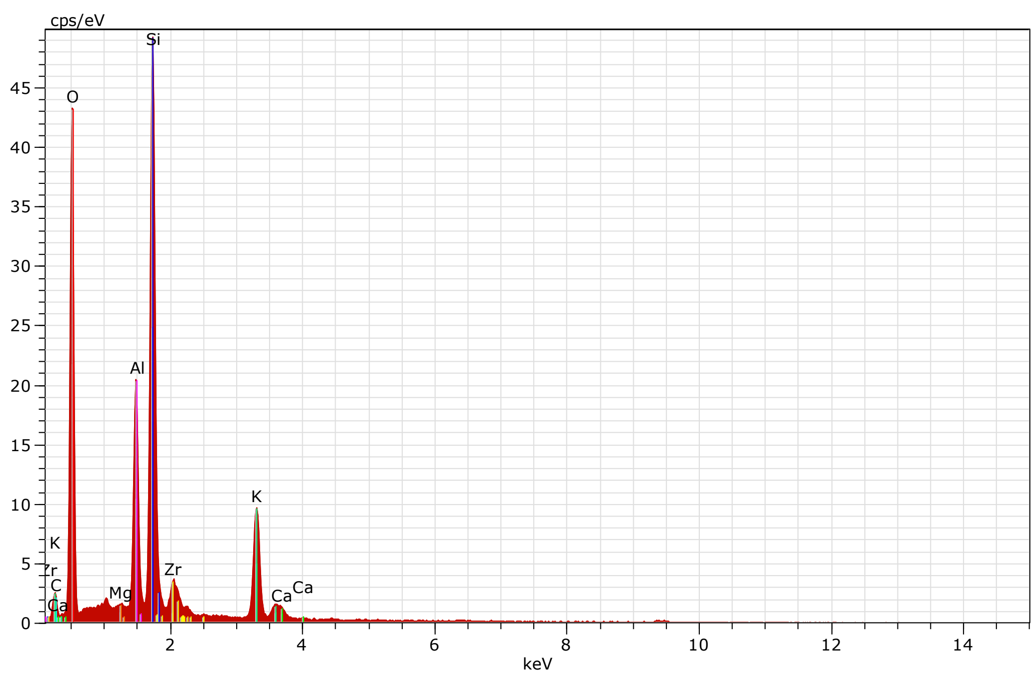


Fig. 4.10: Relative chemical composition for specimen with NS 0.6% b.w.c

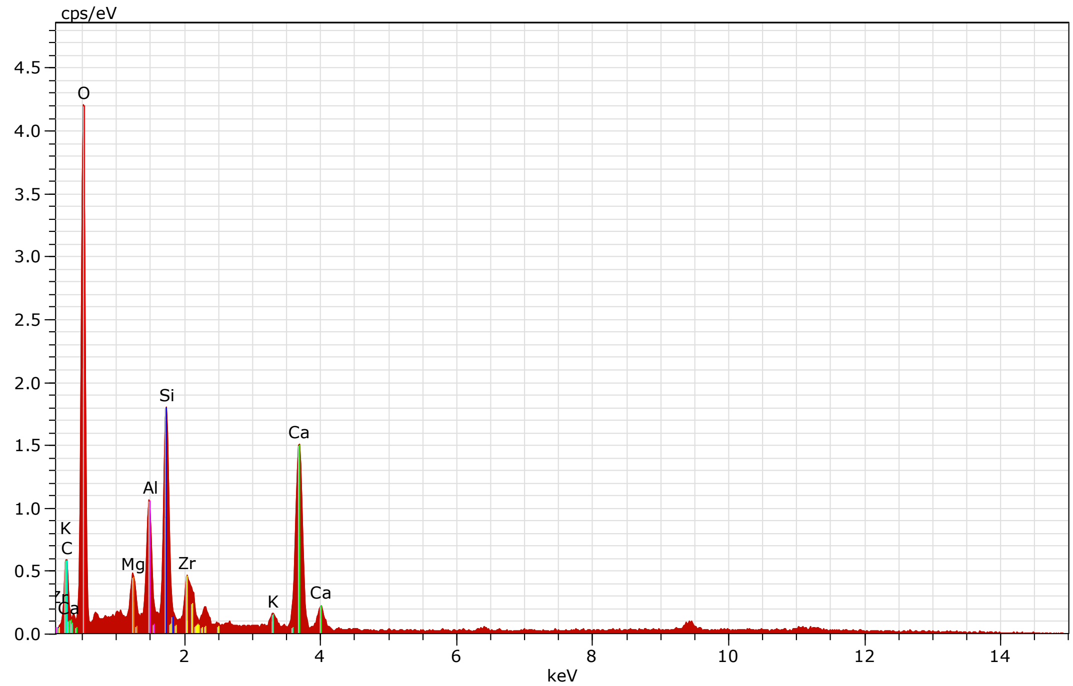


Fig. 4.11: Relative chemical composition for specimen with NS 1% b.w.c

4.4.1Comparison of FESEM micrographs

Fig. 4.4 shows the FESEM micrograph of concrete specimen without nanosilica (NS). In this figure it can be clearly seen that the C-S-H gel is scattered with lots of empty spaces in between the lumps. The lumps can be $Ca(OH)_2$ which weakens the Interfacial Transition Zone (ITZ) hence affecting the strength. The microstructure looks to contain mainly amorphous substances.

Fig. 4.5 shows the FESEM micrograph of concrete specimen with NS 0.3% b.w.c. Here we can see a better packed microstructure but again large lumps of possibly $Ca(OH)_2$ crystals surrounded connected by needle like structures are found which is generally seen in plain concrete. The NS particles occupying the pores in C-S-H gel gives the compact structure but are not sufficient in amount to react with $Ca(OH)_2$ and produce C-S-H gel.

Fig. 4.6 shows the FESEM micrograph of concrete specimen with NS 0.6% b.w.c. A uniform microstructure with very little void can be seen. The absence of $Ca(OH)_2$ crystals indicates that NS has reacted with $Ca(OH)_2$ and converted it into C-S-H gel.

Fig. 4.7 shows the FESEM micrograph of concrete specimen with NS 1% b.w.c. The microstructure is very dense and many crystalline lumps can be observed. These lumps indicates the agglomeration of Nano SiO_2 particles which make the structure crystalline and hence enhances the strength.

4.4.2 Comparison of Chemical Composition of the Specimen

Fig. 4.8 shows the relative chemical composition of concrete specimen without nanosilica (NS).

High concentration of calcium is due to the formation of $Ca(OH)_2$ crystals which weakens the ITZ.

Fig. 4.9 shows the relative chemical composition of concrete specimen with NS 0.3% b.w.c. A high concentration silicon and low concentration of calcium and oxygen shows that silica has got into the structure but hasn’t reacted with the $Ca(OH)_2$ to produce C-S-H gel of which calcium occupies a good portion. These silica particles occupy the pores in the gel and make the microstructure uniform.

Fig. 4.10 shows the relative chemical composition of concrete specimen with NS 0.6% b.w.c. This figure looks contradicting due to high percentage of silica and low percentage of calcium. A good percentage of oxides can be due to the reaction of silica with $Ca(OH)_2$ which produces C-S-H gel.

Another explanation to the increase in strength can be due to the availability of sufficient silica to make the microstructure denser and uniform.

Fig. 4.11 shows the relative chemical composition of concrete specimen with NS 1% b.w.c. The high percentage of oxygen and comparable amount of Ca and Si shows a good reaction between silica and $Ca(OH)_2$ to produce C-S-H gel and hence an increase in strength is observed.

CHAPTER 5: CONCLUSION AND DISCUSSION

5.1 CONCLUSION

From the test results, the SEM micrographs and the relative chemical composition of the specimen a number of conclusions can be drawn. These conclusions are justified in the next section. The conclusions drawn are:

i. From the compressive strength results, it can be observed that increase in compressive strength of concrete is observed on addition of a certain minimum quantity of Nano SiO_2. The increase in strength is maximum for NS 1% b.w.c and least for NS 0.3% b.w.c.

ii. On addition of Nano SiO_2 there is a substantial increase in the early-age strength of concrete compared to the 28 day increase in strength.

iii. The UPV test results show that the quality of concrete gets slightly affected on addition of Nano SiO_2 but the overall quality of concrete is preserved.

iv. The FESEM micrograph shows a uniform and compact microstructure on addition of Nano-SiO_2.

5.2 DISCUSSION

i. The increase in compressive strength can be attributed to the filling of voids in the microstructure by the Nano SiO_2 particles which prevents the growth of $Ca(OH)_2$ crystals. In addition to it the nano silica reacts with calcium hydroxide crystals converting them into C-S-H gel. The reduction in the $Ca(OH)_2$ content is the reason for increase in compressive strength of concrete.

ii. $Ca(OH)_2$ crystals are present in the Interfacial Transition Zone (ITZ) which is between the aggregates and the hardened cement paste. Nano SiO_2 reacts with these crystals and decreases their concentration, hence, strengthen the ITZ. Due to lesser concentration Nano SiO_2 are consumed in the reaction and hence the increase in strength is inhibited with time.

iii. A study of relevant papers show that concrete blended with Nano SiO_2 sets quicker compared to normal concrete. Since, the mix design is carried out without the aid of supper-plasticizers, the mix dried up fast which affected the compaction of the mix using mechanical vibration. Lumps of the mix could be seen during the mixing of concrete. With increase in percentage of Nano SiO_2 the compaction gets tougher. This is the reason for degradation in its quality. It is advisable to use superplasticizers with nano silica.

iv. The Nano SiO_2 added to the mix filled up the pores in between the C-S-H gel, hence, making the microstructure more compact and uniform.

5.3 LIMITATIONS OF THE WORK

The current work has many limitations which are mentioned below:

i. The percentage of nano silica is restricted to 1% due to workability issues which does not give a complete idea about the maximum amount of nano silica that can be added to get an increase in strength.

ii. Without the use of super plasticizers a proper compaction of the concrete was hindered.

iii. 7 day FESEM micrographs are unavailable which could have given a better idea about the early-age increase in strength.

5.4 SCOPE FOR FUTURE RESEARCH

Although a lot of work has been carried out involving the use of nano silica in concrete, a proper understanding has not been developed. In future, the size effects of nano silica can be studied in detail. A detailed study of the microstructure at specific intervals throughout a year can give a very good idea about the reactions taking place in the concrete. Looking at the price of the nano silica new methods can be designed for its production at a low cost.

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APPENDIX

The report of the test conducted to determine the size of the nanoparticle using Particle Size Analyser.

Size Distribution Report by Intensity

v2.1

Malvern

Sample Details

Sample Name:	nanosio2 1
SOP Name:	mansettings.nano
General Notes:	

File Name:	19 apr 15.dts	**Dispersant Name:**	Water
Record Number:	20	**Dispersant RI:**	1.330
Material RI:	1.59	**Viscosity (cP):**	0.8872
Material Absorbtion:	0.01	**Measurement Date and Time:**	Wednesday, April 29, 2015 9:...

System

Temperature (°C):	25.0	**Duration Used (s):**	120
Count Rate (kcps):	90.3	**Measurement Position (mm):**	4.65
Cell Description:	Disposable sizing cuvette	**Attenuator:**	11

Results

			Size (r.nm):	% Intensity	Width (r.nm):
Z-Average (r.nm):	1117	**Peak 1:**	236.6	100.0	39.76
PdI:	1.000	**Peak 2:**	0.000	0.0	0.000
Intercept:	1.15	**Peak 3:**	0.000	0.0	0.000

Result quality : Refer to quality report